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| NASA CASE NO. | LAR-13,597-100 |
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| PRINT FIG. | 1 |

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LaRC

N87-23713

FEOCEDURE TO DETECT A LIQUID-SCIID INTERFACE
LURING CHYSTAL GEORTH IN A REIDGEAN FURNACE
Patent Application (NASA) 10 p Avail: Unclas
NTIS EC A02/MF A01 CSCL 07D G3/25 0076861

(NASA-Case-LAB-13597-1-CU) AFFAFATUS AND

APPARATUS AND PROCEDURE TO DETECT A LIQUID-SOLID INTERFACE DURING CRYSTAL GROWTH IN A BRIDGMAN FURNACE

This invention relates to a method and apparatus for detecting a liquid-solid interface of a substance. In particular, the invention detects the liquid-solid interface of a semiconductor crystal grown in a Bridgman furnace.

As illustrated in the drawings, a substance, such as germanium, in a Bridgman furnace 10 is irradiated by solid-penetrating energy from a radiation source 12. The solid-penetrating energy is detected by a radiation detector 14. Differences in the intensity of the detected energy indicate the shape of the liquid-solid interface. In the case of germanium, X-rays can be used as the solid-penetrating energy, while in the case of a substance such as lead tin telluride, gamma rays are preferable.

The invention enables detection of the shape of the liquidsolid interface of a semiconductor substance during growth of a crystal formed from the substance. Variations in several characteristics of semiconductor crystals are believed to be related to the shape of the liquid-solid interface, but previously there was no means to detect the shape of the liquidsolid interface during growth of the crystal.

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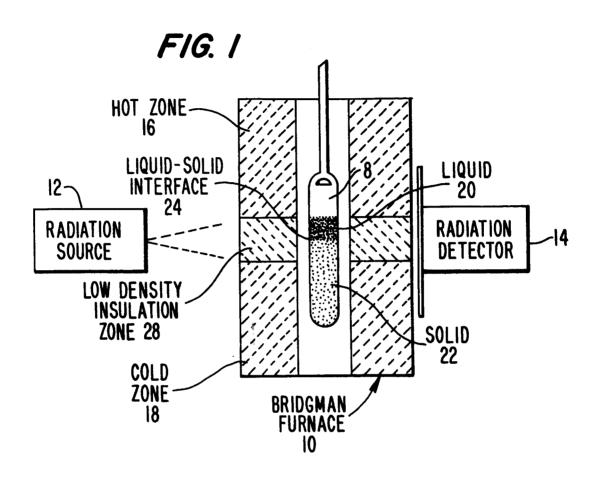


FIG. 2B FIG. 2A HOT ZONE HOT ZONE -16 16-LIQUID LIQUID 1050°C 1010°C **FID**UCIARY FIDUCIARY 26 26 MARK MARK LIQUID LIQUID--20 20 28 28 **INTERFACE** 24 24 INTERFACE -22 22-SOLID SOLID 790°C 815°C -18 18 -SOLID SOLID COLD ZONE COLD ZONE

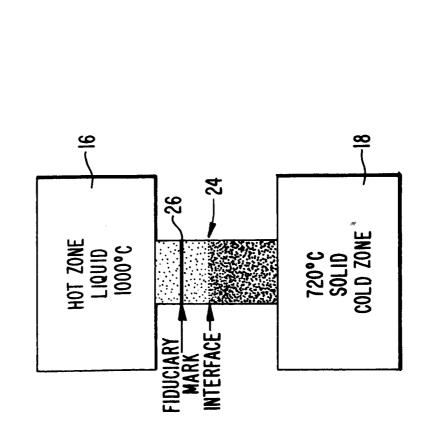
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DETECT A LIQUID-SOLID INTERFACE DURING
CRYSTAL GROWTH IN A BRIDGMAN FURNACE

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F16.34



-INTERFACE

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765°C SOLID COLD ZONE

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F16.3B

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HOT ZONE LIQUID 1000°C Patrick G. Barber et al

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Title of the Invention

APPARATUS AND PROCEDURE TO DETECT A LIQUID-SOLID INTERFACE
DURING CRYSTAL GROWTH IN A BRIDGMAN FURNACE

Origin of the Invention

The invention described herein was made in the performance of work under a NASA Contract and is subject to the provisions of Section 305 of the National Aeronautics and Space Act of 1958, Public Law 85-568 (72 Stat. 435; 42 USC 2457).

10 Background of the Invention

Field of the Invention

The present invention is related to a method and apparatus for detecting a liquid-solid interface and, more particularly, to a method and apparatus which can be used to detect the liquid-solid interface of crystals formed from germanium or lead tin telluride in a Bridgman furnace.

Description of the Related Art

It has long been known that the shape of the liquidsolid interface during solidification of semiconductor

20 crystals significantly alters the final properties of the
crystal. Properties which may be affected include impurity
distribution, defect generation, and effects caused by the
flow patterns in a system with radial temperature and
solutal gradients. However, heretofore, all investigation

25 of the correlation between interface shape and these

effects has required that the analysis be performed after the crystal growth is completed, because there has been no known way to detect the shape of the liquid-solid interface during crystal growth.

There are known techniques for detecting gas-liquid and gas-solid interfaces of a substance, e.g., U.S. Patents 3,668,392 to Bajek et al. and 4,358,682 to Telfer et al. and also for detecting interfaces between dissimilar substances, such as between molten metal and slag as taught by U.S. Patent 4,433,242 to Harris et al. However, in addition to not detecting a liquid-solid interface of a single substance, all three of these patents are directed to detecting the location of the interface rather than its shape.

15 Summary of the Invention

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An object of the present invention is to detect a liquid-solid interface of a single substance.

Another object of the present invention is to provide a two-dimensional view of a liquid-solid interface of a substance.

A further object is to detect growth of a crystal in a Bridgman furnace.

A yet further object of the invention is to control a Bridgman furance to provide a desired liquid-solid interface shape and position of a substance by first viewing the radiation image thereof.

The above objects are attained by detecting a liquidsolid interface of a substance using a method which includes the steps of irradiating the substance with a solid-penetrating energy output by a radiation source and detecting differences in intensity of the solid-penetrating energy in a detection area having at least two dimensions. The substance is disposed between the radiation source and the detection area. A first level of intensity results from passage through a liquid portion of the substance and a second level of intensity results from passage through a solid portion of the substance.

Preferably, gamma rays or X-rays are used as the solidpenetrating energy which irradiates the substance. The solid portion of the substance may be a crystal which is being produced by controlling temperature in the area of the liquid-solid interface.

The steps described above may be used to detect a liquid-solid interface formed during growth of a crystal using energy direction means for directing solid-penetrating energy onto a first side of the crystal at a first area including a liquid-solid interface and detection means for detecting intensity of the solid-penetrating energy in a second area facing a second side, opposite the first side of the crystal.

These objects, together with other objects and advantages which will be subsequently apparent, reside in the details of construction and operation as more fully hereinafter described and claimed, reference being had to the accompanying drawings forming a part hereof, wherein like reference numerals refer to like parts throughout.

Brief Description of the Drawings

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FIG. 1 is a schematic diagram of an apparatus according to an embodiment of the present invention;

FIGS. 2A and 2B are illustrative of the image formed on X-ray film by X-rays which have passed through a Bridgman furnace in which a germanium crystal is being formed; and

FIGS. 3A and 3B are illustrative of the image formed by detecting gamma rays which have passed through a Bridgman furnace in which a lead tin telluride crystal is being grown.

Description of the Preferred Embodiments

As illustrated in FIG. 1, a substance 8 is supplied to a Bridgman furnace 10, which was constructed by the inventors, to grow a crystal. A radiation source 12 irradiates the substance 8 from a first (left) side of the Bridgman furnace 10. A radiation detector 14 on a second (right) side of the Bridgman furnace detects differences in intensity of the solid-penetrating energy output by the radiation source 12. For example, when germanium is used 10 as the substance 8, X-rays may be used as the solidpenetrating energy, since germanium has a relatively low atomic number and is thus penetrated by X-rays. case, the radiation source 12 may be a Model SPX 160 X-ray tube, manufactured by Automation Industries of Danbury, Connecticut, and the radiation detector 14 could include X-15 ray sensitive film, such as Kodak Type M Radiographic film, manufactured by Eastman Kodak Company of Rochester, New York. The X-ray sensitive film produces a record of the radiation intensity in a two-dimensional area on the second 20 side of the Bridgman furnace.

A Bridgman furnace is illustrated in FIG. 2A which has a hot zone 16 producing liquid germanium at a temperature of 1040°C and has a cold zone 18 set at 790°C. Theoretically, there should be an X-ray density difference between the liquid germanium 20 and solid germanium 22 of 25 approximately four percent. As a result, an interface 24 is formed as illustrated. In the illustrated embodiment, a fiduciary mark 26 is formed to provide a reference point by, e.g., a ceramic coating on shuttle tile material used to provide an insulation zone 28 in the portion of the 30 Bridgman furnace in which the crystal is grown. The effect of increasing the temperature of the hot 16 and cold 18 zones is illustrated in FIG. 2B where the interface 24 between the liquid 20 and solid 22 is noticeably lower.

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In the case of substances that are high in atomic number, such as lead tin telluride, gamma rays can be used as a solid-penetrating energy to produce images such as those illustrated in FIGs. 3A and 3B for different cold 18 zone temperature settings. In this case, the radiation source 12 may be an Irridium 192, 0.1 inch x 0.1 inch right cylinder, manufactured by Gamma Industries of Baton Rouge, Louisiana, and the radiation detector 14 could be Kodak Type M Radiographic film. When lead tin telluride is grown vertically downward, i.e., hot-on-the-bottom, it develops a central void along its length. The development of such a void can be followed by taking a series of "pictures" using gamma radiation.

The many features and advantages of the present 15 invention are apparent from the detailed specification, and thus, it is intended by the appended claims to cover all such features and advantages of the device which fall within the true spirit and scope of the invention. Further, since numerous modifications and changes will readily occur to those skilled in the art, it is not 20 desired to limit the invention to the exact construction and operation illustrated and described. Accordingly, all suitable modifications and equivalents may be resorted to falling within the scope and spirit of the invention. For example, it is expected that the images provided can be 25 improved using computer aided image enhancement and that an electronic array can be used as the radiation detector 14 in place of X-ray sensitive film.

What is claimed is:

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PATENT

APPARATUS AND PROCEDURE TO DETECT A LIQUID-SOLID INTERFACE DURING CRYSTAL GROWTH IN A BRIDGMAN FURNACE

Abstract of the Disclosure

A method of detecting a liquid-solid interface of a substance includes irradiating the substance with a solid penetrating energy and detecting differences in intensity of the solid-penetrating energy which passes through the substance. A first level of intensity results from passage through a liquid portion of the substance and a second level of intensity results from passage through a solid portion of the substance.